इंटरनेट मानक

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"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
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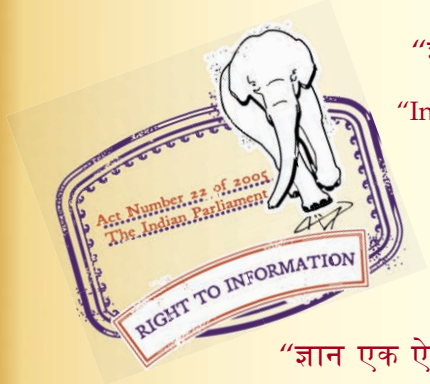
IS 8982 (1991): Ready mixed paint, finishing air drying for war equipment [CHD 20: Paints, Varnishes and Related Products]



"ज्ञान से एक नये भारत का निर्माण"
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"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

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IS 8982 : 1991
(Reaffirmed - 2012)

भारतीय मानक

युद्ध उपस्करों के लिए तैयार मिश्रित रोगन, फिनिश देने वाले, हवा में सूखने वाले — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

READY MIXED PAINT, FINISHING, AIR-DRYING FOR WAR EQUIPMENT — SPECIFICATION

(First Revision)

UDC 667.688 : 623.4

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1991

Price Group 5

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Paints and Allied Products Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1978. It was made on a request from Ministry of Defence and was based on JSS 8010-15 'Specification for paint, ready for use, finishing, war equipment, air drying, brushing/spraying, olive green, white and buff light, Indian Standard Colour No. 358'. During the last decade, the technology advancement has taken place and in this revision, the values of some of the requirements such as wet opacity, gloss, fineness of grind, etc, have been quantified. New requirements of accelerated storage/stability test have been added to align it fully with the revised defence specifications.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 2 APRIL 2006
TO
IS 8982 : 1991 READY MIXED PAINT, FINISHSING, AIR-DRYING FOR WAR EQUIPMENT — SPECIFICATION

(First Revision)

(*Page* 1, *clause* **4.2,** *line* 2) — Delete the words 'olive green'.

(CHD 20)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 1 JANUARY 1999
TO
IS 8982 : 1991 READY MIXED PAINT, FINISHING, AIR-DRYING FOR WAR EQUIPMENT— SPECIFICATION

(First Revision)

[*Page* 2, *Table* 1, *Sl No.* 4(c), *col* 2] — Substitute 'Fineness of grind, micron,

[*Page* 2, *Table* 1, *Sl No.* 4(c), *col* (3) — Substititute '40' *for* '5'.

[*Page* 2, *Table* 1, *Sl No,* 6, *col* 3 *line* 2] — Substitute 'Bare metal shall not be visible through the scrasch after 48 hours' for the existing matter.

[*Page* 2, *Table* 1. *Sl No.* 6, *col* 5] — Substitute 'IS 101 (Part 5/Sec 2) : 1988' *for* 'IS 101 (Part 5/Sec 1) : 1988'.

[*Page* 2, *Table* 1, *Sl No.* 9. *col* 2] — Substitute 'Resistance to lubricating oil [*see* IS 13656 : 1993 Internal combustion engine crankeses oils (gasoline and diesel)]' for the existing matter.

[*Page* 2, *Table* 1, *Sl No.* 9, *col* 5] — Substitute '4 of IS 101 (Part 7/Sec 2)' *for* *IS 10l (Part 7/Soc 2)'.

[*Page* 5. *Annex* B, *clouse* **B-1.7**, *line* 2] — Substitute 'Specific gravity 1.8' *for* 'Relative density 1 percent'.

(CHD 31)

Reprography Unit, BIS, New Delhi, India

Indian Standard

READY MIXED PAINT, FINISHING, AIR-DRYING FOR WAR EQUIPMENT — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for the material commercially known as ready mixed paint, finishing, air drying, for war equipment. The material is used for protection and maintenance of wooden and metal parts of war equipment including vehicles, machine guns, bridging equipment, etc and also on POL containers as exterior petrol resisting paint.

1.1.1 The material shall normally be applied over a suitable priming paint but formulation shall be such as to provide reasonable camouflaging and protection if applied direct on metal and wooden surfaces.

2 REFERENCES

The Indian Standards listed in Annex A are necessary adjuncts to this standard.

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 1303 : 1983 shall apply.

4 REQUIREMENTS

4.1 General

The material shall be supplied in brushing consistency but shall be suitable for application by spraying after thinning with petroleum hydrocarbon solvent, low aromatic, 145/205 grade (*see* IS 1745 : 1978).

4.2 Colours

The colour of the material shall be white or scamic (olive green) Indian Standard Colour No. 294 or light buff Indian Standard Colour No. 358, as required. It shall be close match to specified colour when tested as per IS 101 (Part 4/Sec 2) : 1989.

4.3 Pigment Composition

The dry pigment content of the material shall be 33 ± 3 percent by mass. In case of olive green paint, the pigment shall contain minimum 6 percent by mass of green oxide of chromium when determined as prescribed in Annex B.

4.4 The material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

Unless otherwise agreed to between the purchaser and the supplier, the paint shall be packed in metal containers conforming to IS 1407 : 1980 and IS 2552 : 1979.

5.2 Marking

Each container shall be marked with the following:

a) Name and colour of the material.

b) Indication of the source of manufacture,

c) Volume of the material,

d) Batch No. or Lot No. in code or otherwise, and

c) Month and year of manufacture.

6 SAMPLING

6.1 Representative samples of the material shall be drawn and treated as prescribed in IS 101 (Part l/Sec 1) : 1986.

6.2 Criteria for Conformity

A lot shall be declared conforming to the requirements of this specification if the test results on the composite sample satisfy the requirements prescribed in **4**.

7 TEST METHODS

7.1 Test shall be conducted according to the methods referred to in col 4 and 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*sec* IS 1070 : 1977) shall be employed in tests.

NOTH — 'Pure chemical' shall mean chemicals that do not contain impurities which affect the results of analysis.

7.3 For match against Indian Standard colour. IS 5 : 1978 shall be used.

Table 1 Requirements for Ready Mixed Paint, Finishing, Air Drying for War Equipment
(*Clauses* **4.4** *and* **7.1**)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	IS No.
(1)	(2)	(3)	(4)	(5)
1.	a) Consistency	Smooth and Uniform		101 (Part 1/Sec 5)
	b) Efflux time by Ford Cup No. 4 at 27 ± 2°C. *Sec*	80-120	—	
	Application	Suitable for application by brushing without appreciable drag on the brush	—	6 of 101 : 1964
3.	Drying time:			
	a) Surface dry	Not less than 1 hour	—	101 (Part 3/Sec 1) : 1986
	b) Hard dry and tack free	Not more than 8 hours		
4.	a) Finish	Smooth and semi-glossy	—	101 (Part 3/Sec 4) : 1987
	b) Gloss value, 45° (Specular Gloss)	31 - 50 units	—	101 (Part 4/Sec 4) : 1988
	c) Fineness of grind (Hegman scale), *Min*	5	—	101 (Part 3/Sec 5) : 1987
5.	Wet opacity, *Min* m^2/IOL (for olive green only)	250		101 (Part 4/Sec 1) : 1988
6.	Scratch hardness (1 500 g)	Bare metal shall not be visible through the scratch	—	101 (Part 5/Sec 1) : 1988
7.	*a) Flexibility and adhesion	No damage or detachment or cracking of the film	—	101 (Part 5/Sec 2) : 1988
	†b) Flexibility and adhesion at 0°C (only for white)	No damage or detachment or cracking of the film	—	—
8.	Protection against corrosion	There shall be not more than very slight change of colour not more than slight superficial rust spotting, no blistering of permanent nature (blistering which does not subside within 24 hours after removal of the painted panel from the paint corrosion cabinet shall be termed as permanent), no brittleness of the paint film and the metal surface underneath the film shall not show any sign of corrosion		‡101 (Part 6/Scc 1) : 1988
9.	Resistance to lubricating oil	No sign of permanent injury	—	101 (Part 7/Sec 2)
10.	Resistance to petrol-benzol mix-	No sign of permanent injury	C	—
11.	Resistance to heat (except white)	No sign of cracking, blistering and appreciable change of colour when stoved at 150°C for 2 hours. In this respect the film shall not be inferior to registered sample		101 (Part 7/Sec 3) : 1990
12.	Water absorption test	Amount of water absorbed shall not exceed 20 mg/cm^2 of painted surface exposed	D	
13.	Resistance to warm water at 45 ± 3°C for 4 hours	No fading or change of colour	E	—
14.	Resistance to salt water	No rust spotting, flaking or peeling or any other deterioration of the paint film when examined at a magnification of 10	F	—
15.	Durability	To pass the test	G	—
16.	Flash point, °C, *Min*	30	—	101 (Part 1/Sec 6) : 1987

Table 1 (*Concluded*)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	IS No.
(1)	(2)	(3)	(4)	(5)
17.	Calcium compounds in dry pigments (as CaO), percent by mass. *Max*	5	H	—
18.	Barium compounds in dry pigments (as $BaS0_4$), percent by mass. *Max*	33	J	—
19.	Water content (if water suspected to be present), percent by mass. *Max*	0 5		101 (Part 2/Sec 1) : 1988
20.	Lead restriction	Not a lead paint	—	28 of 101 : 1964
21.	Mass in kg/10 litres, *Min*	11 0	—	101 (Part 1/Sec 7) : 1987
22.	Accelerated storage stability test	To pass the test	K	—
23.	Keeping properties, *Min*	The material shall not cake hard inside the containers and shall retain properties for one year from the date of delivery	—	101 (Part 6/Sec 2) : 1989

*The panel after application shall be stoved at 100°C for one hour after initial drying of 24 hours. The test shall be carried out after the panels are allowed to stand at room temperature not below 15°C for a further period of 24 hours.

†The panel shall initially be air dried at room temperature of not below 15°C for seven days and thereafter shall be kept along with the bend test apparatus at 0°C for 2 hours

‡Exccpt for duration of the test period which shall be 10 days.

ANNEX A

(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*	*IS No.*	*Title*
5 : 1978	Colours for ready mixed paints and enamels (*third revision*)	101 (Part 1/Sec 5)	Methods of sampling and test or paints, varnishes and related products : Part 1 Test on liquid paints (general and physical). Section 5 Consistency
101 : 1964	Methods of test for ready mixed paints and enamels (*second revision*)		
101 (Part I/Sec 1) : 1986	Methods of sampling and test for paints, varnishes and related products : Part 1 Test on liquid paints (general and physical). Section 1 Sampling (*third revision*)	101 (Part 1/Scc 6) : 1986	Methods of sampling and test for paints, varnishes and related products : Part 1 Test on liquid paints (general and physical), Section 6 Flash point (*third revision*)
101 (Part 1/Sec 3) : 1986	Methods of sampling and test for paints, varnishes and related products : Part 1 Test on liquid paints (general and physical), Section 3 Preparation of panels (*third revision*)	101 (Part 1/Sec 7) : 1987	Methods of sampling and test for paints, varnishes and related products : Part 1 Test on liquid paints (general and physical), Section 7 Mass per 10 litres (*third revision*)

IS No.	*Title*
101 (Part 2/Sec 1) : 1988	Methods of sampling and test for paints, varnishes and related products : Part 2 Test on liquid paints (chemical examinations). Section 1 Water content
101 (Part 3/Sec 1) : 1986	Methods of sampling and test for paints, varnishes and related products : Part 3 Tests on paint film formation, Section 1 Drying time (*third revision*)
101 (Part 3/Sec 4) : 1987	Methods of sampling and test for paints, varnishes and related products : Part 3 Tests on paint film formation, Section 4 Finish (*third revision*)
101 (Part 3/Sec 5) : 1987	Methods of sampling and test for paints, varnishes and related products : Part 3 Tests on paint film formation, Section 4 Fineness of grind (*third revision*)
101 (Part 4/Scc 1) : 1988	Methods of sampling and test for paints, varnishes and related products : Part 4 Optical tests on paint films, Section 1 Opacity (*third revision*)
101 (Part 4/Sec 2) : 1989	Methods of sampling and test for paints, varnishes and related products : Part 4 Optical test on paint films, Section 2 Colour (*third revision*)
101 (Part 4/Sec 4) : 1988	Methods of sampling and test for paints, varnishes and related products : Part 4 Optical tests on painr films, Section 4 Gloss (*third revision*)
101 (Part 5/Sec 1) : 1988	Methods of sampling and test for paints, varnishes and related products: Part 5 Mechanical test on paint films, Section 1 Hardness test (*third revision*)
101 (Part 5/Sec 2) : 1988	Methods of sampling and test for paints, varnishes and related products: Part 5 Mechanical test on paint films. Section 2 Flexibility and adhesion (*third revision*)
101 (Part 6/Sec 1) : 1988	Methods of sampling and test for paints, varnishes and related products : Part 6 Durability tests. Section 1 Resistance to humidity under conditions of condensation (*third revision*)
101 (Part 6/Sec 2) : 1989	Methods of sampling and test for paints, varnishes and related products : Part 6 Durability tests. Section 2 Keeping properties (*third revision*)
101 (Part 7/Sec 2)	Methods of sampling and test for paints, varnishes and related products : Part 7 Environmental tests on paint films, Section 2 Resistance to liquids (*third revision*)
101 (Part 7/Sec 3) : 1989	Methods of sampling and test for paints, varnishes and related products : Part 7 Environmental tests on paint films. Section 3 Resistance to heat (*third revision*)
1303 : 1983	Glossary of terms relating to paints (*second revision*)
1407 : 1980	Round paint tins (*second revision*)
2552 : 1979	Steel drums (galvanized and ungalvanized) (*second revision*)

ANNEX B

(*Clause* 4.3)

DETERMINATION OF GREEN OXIDE OF CHROMIUM

B-0 GENERAL

Chromium oxide (Cr_2O_3) content of the pigment is estimated from acid insoluble chromium compounds. The method involves the estimation of total chromium compounds as Cr_2O_3 in pigment of the paint.

B-1 REAGENTS

B-1.1 Sodium Peroxide

B-1.2 Sulphuric Acid

concentrated.

B-1.3 Potassium Permanganate Solution

5 percent (*m/v*).

B-1.4 Silver Nitrate Solution

10 percent (*m/v*).

B-1.5 Ammonium Persulphate

B-1.6 Hydrochloric Acid

Dilute.

B-1.7 Phosphoric Acid

Relative density 1 percent.

B-1.8 Ferrous Ammonium Sulphate Solution

approximately 0.1 N.

B-1.9 Standard Potassium Permanganate Solution

0.1 N.

B-2 ESTIMATION OF CHROMIUM COMPOUNDS IN PAINT

B-2.1 Procedure

Weigh accurately about 1 to 2 g of the paint in a nickel crucible and ignite over a gas flame. Fuse it with 5 g of sodium peroxide over a gas flame, keeping the molten mass at low red heat for 5 minutes. Dissolve the cooled fusion cake in the crucible in 200 ml of warm water and transfer the rinsings to a beaker. Add concentrated sulphuric acid to adjust 10 percent acidity in the solution. Add 4 to 6 drops of 5 percent potassium permanganate solution and 1 to 2 ml of silver nitrate solution. Heat to boil. Add ammonium persulphate till pink colour of potassium permanganate reappears. Add dilute hydrochloric acid just to destroy the pink colour. Boil to expel chlorine. Cool and add 5 ml of phosphoric acid. Add exactly 50 ml of ferrous ammonium sulphate solution with vigorous stirring. A deep green colour develops when reduction is complete. Titrate the excess of ferrous ammonium sulphate solution with standard potassium permanganate solution, taking the first faint darkening of the clear green colour as the end point. Run a blank, starting from peroxide fusion, in the same manner and at the same time. Note the volumes of potassium permanganate solution required in both cases.

B-3 ESTIMATION OF ACID SOLUBLE CHROMIUM COMPOUNDS IN PIGMENT

B-3.1 Procedure

Weigh accurately about 0.5 to 1 g of the dried pigment of the material sample and digest it in 200 ml of water in a covered beaker. Add 25 ml of sulphuric acid. Boil for 10 minutes, cool and filler. Then proceed as prescribed in **B-2.1**.

B-4 CALCULATION

B-4.1 Chromium oxide in both the cases as prescribed in **B-2.1** and **B-3.1** is calculated as follows:

$$\text{Chromium oxide (as } Cr_2O_3\text{), percent by mass} = \frac{(A - B) \times N \times 2{\cdot}534}{M}$$

where

A = volume in ml of potassium permanganate solution required for blank titration,

B = volume in ml of potassium permanganate solution required in the assay.

N = normality of potassium permanganate solution, and

M = mass in g of the sample taken for the test.

B-4.2 Calculate percent total Cr_2O_3 content of total chromium in pigment as follows:

$$\text{(Calculated chromium oxide content in paint)} \times \frac{100}{\text{(Percent pigment in the sample of paint)}}$$

B-4.3 Calculate acid insoluble chromium oxide as follows:

$$\text{Acid insoluble chromium (as } Cr_2O_3\text{) in pigment, percent by mass} = X - Y$$

where

X = total chromium (as Cr_2O_3) calculated in **B-4.2**, and

Y = acid soluble chromium oxide as determined in **B-3.1**.

ANNEX C

(*Table* 1, *Item* 10)

TEST FOR RESISTANCE TO PETROL-BENZOL MIXTURE

C-1 REAGENTS

C-1.1 Benzol

pure.

C-1.2 Petroleum Ether

boiling range 60 to 80°C, aromatic free.

C-2 PROCEDURE

C-2.1 Take a tin plate panel 150 mm × 50 mm × 0.315 mm free from surface imperfections, roughen with No. 0 abrasive paper. Apply the paint by brushing in a single coat to give a dry film mass as specified in IS 101 (Part 3/Sec 4) : 1987. Allow the paint film to air-dry for seven days followed by keeping it for 24 h under laboratory drying conditions. Then immerse in a mixture of 5 parts by volume of benzol and 95 parts by volume of petroleum ether for 5 minutes at room temperature not below 15°C. Examine the paint film 30 minutes after removal of the panel from the solvent mixture.

C-2.2 The material shall be taken to have passed the requirement of this test if the paint film does not show any sign of permanent injury and withstands the scratch hardness test.

ANNEX D

(*Table* 1, *Item* 12)

TEST FOR WATER ABSORPTION

D-1 REAGENTS

D-1.1 Paraffin Wax and Beeswax (1 : 1) Mixture

molten.

D-2 PROCEDURE

D-2.1 Take a minimum of 3 panels of reasonably straight grained and well seasoned (to a moisture content of not more than 12 percent) chirwood (*Pinus longifolia*) approximately 100 mm × 100 mm × 9 mm size, round edges and corners with No. 0 abrasive paper and clean it free from dust and extraneous matter. Apply one coat of the material by brushing so as to completely paint the panels including edges and allow to dry for 24 hours at room temperature not below 15°C.

D-2.2 Apply two further coats of the paint in a similar manner and allow the panels to dry for 48 hours after the final coat. Seal the edges and corners of the painted panels to a depth of 6 mm all round, by dipping in molten paraffin and beeswax mixture.

D-2.3 Weigh the panel and then completely immerse them in a water-bath maintained at 27 ± 2°C and allow to remain immersed in water for 7 days. Remove the panels from water and absorb the excess moisture by blotting paper and weigh them again in such a manner that the time interval between the withdrawal of panels from water-bath and the weighment is not less than 3 minutes and not more than 5 minutes. Note the gain in mass and divide by the total area of the paint film on both sides of the panels not coated with wax-mixture to calculate the results.

ANNEX E

(*Table* 1, *Item* 13)

TEST FOR RESISTANCE TO WARM WATER

E-1 PROCEDURE

Take a mild steel plate, 150 mm × 100 mm × 1.25 ram free from surface imperfections and roughen with No. 0 abrasive paper. Paint the panel and keep it under drying conditions prescribed in C-2. Immerse the panel in water maintained at 45 ± 2°C for 4 hours. Remove the panel, keep at room temperature for 1 hour and examine the dried paint film for any fading or change in colour.

ANNEX F

(*Table* 1, *Item* 14)

TEST FOR RESISTANCE TO SALT WATER

F-1 REAGENTS

F-1.1 Calcium Sulphate

anhydrous.

F-1.2 Magnesium Chloride

anhydrous.

F-1.3 Magnesium Sulphate

anhydrous.

F-1.4 Sodium Chloride

F-2 PROCEDURE

F-2.1 Prepare a panel of mild steel of approximately 150 mm × 150 mm × 1.25 mm size as prescribed in IS 101 (Part 1/Sec 3) : 1986, and paint with the material by brushing as prescribed in **C-2.1.** Allow the film of the material to air-dry for 24 hours at room temperature not below 15°C. Apply another coat of the material over it and allow the panel to air-dry for 48 hours. Expose the panel to the following cycle:

a) One hour immersion in salt water of the following composition:

1) Calcium sulphate, anhydrous	1.3 g
2) Magnesium chloride, anhydrous	2.6 g
3) Magnesium sulphate, anhydrous	1.7 g
4) Sodium chloride	21.4 g
5) Water to make	1 000 ml

b) Three hour exposure outside at an angle of 45° facing south.

c) One hour refrigeration at 4 ± 2°C.

d) Exposure outside at 45° facing south till the commencement of the next cycle.

F-2.2 Repeat the cycle 12 times exposing the same side of the panel during each cycle. Examine the exposed side for signs of breakdown of the paint film.

ANNEX G

(*Table* 1, *Item* 15)

TEST FOR DURABILITY

G-1 PREPARATION OF PANEL

Prepare the panel as described in **F-2.1.**

G-2 PROCEDURE

Expose the painted panels, in duplicate, outdoors for 6 months at an angle of 45° facing south. At the end of this period examine the test panels under × 10 magnification. The material shall be taken to have passed the durability test if there is no sign of checking brittleness or other impairment of film integrity, not more than a trace of chalking and no appreciable fading/darkening or change of colour and loss of gloss. In this respect the sample shall not be inferior to the registered sample when tested in the similar manner and at the same time.

NOTE — Provisional acceptance may be given on one month exposure on separate set of painted panels as follows, but the durability of the material be taken only after 6 months exposure as above. The painted panel shall be exposed outdoors for 25 days at an angle of 45° facing south. At the end of this period the panel shall be subjected to a tine spray of water for 6 hours at a temperature not above 30°C. (A suitable spray is provided by a water supply at a pressure of approximately 13.7 kPa through a rose.) The treatment shall be carried out daily for six days. The test panels shall be exposed at an angle of 45° facing south between treatment.

ANNEX H

(*Table* 1, *Item* 17)

DETERMINATION OF CALCIUM COMPOUNDS

H-1 REAGENTS

H-1.1 Dilute Hydrochloric Acid

1 : 1 (*v/v*).

H-1.2 Ammonium Hydroxide

20 percent.

H-1.3 Hydrogen Sulphide

gas.

H-1.4 Ammonium Oxalate Solution

saturated.

H-1.5 Standard Potassium Permanganate Solution

0.1 N.

H-1.6 Concentrated Nitric Acid

H-1.7 Ammonium Chloride

H-2 PROCEDURE

H-2.1 Weigh accurately about 0.5 g of the extractedpigment in a 250-ml beaker. Add 50 ml of dilute hydrochloric acid and boil gently for about 5 minutes. Dilute the solution to about 200 ml with water and boil again for 2 minutes. Place the beaker on a water-bath and boil for about 2 to 3 hours. Filter the hot solution through a filter paper (Whatman No. 30 or equivalent) and wash the residue with hot water till the filtrate is free from chloride and lead ions.

H-2.2 Treat the filtrate obtained in **H-2.1** with a few drops of dilute nitric acid and boil. Add about 5 g of ammonium chloride and make the solution alkaline with ammonium hydroxide. Boil for 15 minutes and filter through a filter paper. Wash the precipitate with water and then pass a stream of hydrogen sulphide gas through the filtrate for 15 minutes. Filter off the sulphides and wash 2 to 3 times with saturated solution of hydrogen sulphide. Boil the filtrate to remove hydrogen sulphide and continue boiling after acidifying the solution. Make the solution alkaline to litmus with ammonium hydroxide and heat to boil. Add sufficient quantity of ammonium oxalate solution and boil for 5 minutes. Allow the beaker with the precipitate to stand for 2 hours on a water-bath. Filter the residue through a filter paper (Whatman No. 30 or equivalent) and wash several times with hot water. Tgnite it in a porcelain crucible to calcium oxide, cool in a desiccator and weigh to constant mass.

H-2.3 Alternatively, wash the residue on the filter paper with hot water until the filtrate is free from oxalate ions. Wash down the residue on the filter paper into the original beaker with hot water and wash the filter paper with dilute sulphuric acid collecting the washings in the beaker. Heat to about 70 to 80°C and titrate with standard potassium permanganate solution. When the end point is reached, put the filter paper on which the precipitate was washed in the beaker and complete the titration by adding further quantity of permanganate solution. (For filter paper not more than two drops of standard potassium permanganate need be required.)

H-3 CALCULATION

H-3.1 Ignition Method

$$\text{Calcium oxide, percent by mass} = 100 \times \frac{M_1}{M_2}$$

where

M_1 = mass in g of calcium oxide obtained, and

M_3 = mass in g of the pigment taken for the analysis.

H-3.2 Titration Method

$$\text{Calcium oxide, percent by mass} = \frac{2{\cdot}8\ V\ N}{M_2}$$

where

V = volume in ml of standard potassium permanganate solution,

N = normality of potassium permanganate solution, and

M_2 = mass in g of the pigment taken for the analysis.

ANNEX J

(*Table* 1, *Item* 18)

DETERMINATION OF BARIUM SULPHATE

J-1 REAGENTS

J-1.1 Sodium Carbonate

anhydrous.

J-1.2 Sodium Carbonate Solution

2 g/l.

J-1.3 Dilute Hydrochloric Acid

1 : 4.

J-1.4 Ammonium Hydroxide

20 percent (*m/m*).

J-1.5 Concentrated Hydrochloric Acid

J-1.6 Nitric Acid

1 : 1 (*v/v*).

J-1.7 Methyl Orange Indicator Solution

Dissolve 0.1 g of methyl orange in 100 ml of water.

J-1.8 Ammonium Sulphate Solution

3 percent.

J-2 PROCEDURE

J-2.1 Weigh about 1 g of extracted pigment in a dry and clean 250-ml beaker. Add 20 ml of dilute nitric acid (1 : 1 by volume). Cover the beaker and digest over hot plate for one hour. Dilute the contents to about 150 ml, filter through filter paper, wash the insoluble residue several times. Ignite the residue in platinum crucible. Cool it and add about 8 g of anhydrous sodium carbonate and cover the crucible. Fuse the mixture over a Meker burner for 40 minutes. The fusion is started with a low flame which is gradually raised to full blast. This precaution is necessary to prevent loss due to overflowing.

J-2.2 Cool and as the melt cools, rotate the crucible so that the fused mass solidifies in a thin layer. This will shorten the time required for leaching. Leach out the fusion with 200 ml of hot water in a 400-ml beaker. Filter through a 15 cm filter paper (Whatman No. 40 or equivalent). Wash several times by decantation, then remove the crucible from the beaker, transfer the insoluble carbonates to the filter and wash with hot sodium carbonate solution, testing after the twelfth washing to be certain that sulphates have been removed completely.

J-2.3 Cover the funnel containing the insoluble carbonates with a watchglass and add hot dilute hydrochloric acid carefully, in small portions at a time to prevent loss, catching the solution in a 600-ml beaker. Add hot dilute hydrochloric acid to the platinum crucible and the beaker in which the leach was made and pour over the filter. Wash the paper with hot water until free from chlorides.

J-2.4 Neutralize this solution with ammonium hydroxide using methyl orange as indicator. Add 0.4 to 0.6 ml of concentrated hydrochloric acid. Dilute to 400 ml with hot water, bring the solution to boiling, and add 25 ml of hot ammonium sulphate solution dropwise with constant stirring to prevent co-precipitation of calcium and magnesium. Transfer the beaker to a warm hot plate and allow to stand for at least 4 hours. Filter on an ignited weighed Gooch crucible, wash with hot water several times by decantation. The beaker should be scrubbed thoroughly to remove any adhering precipitate. Continue the washing until free of chlorides. Ignite the crucible in a muffle furnace for 35 minutes at 850 C. Cool in a desiccator and weigh. Make a blank determination in a similar manner with an equal amount of sodium carbonate and other reagents.

J-3 CALCULATION

$$\text{Barium sulphate, percent by mass} = 100 \times \frac{(A - B)}{M}$$

where

A = mass in g of the precipitate with the material,

B = mass in g of the precipitate in the blank determination, and

M = mass in g of the material taken for the test.

ANNEX K

(*Table* 1, *Item* 22)

DETERMINATION OF ACCELERATED STORAGE STABILITY

K-1 The paint sample is stored in a closed container and kept at 60°C for 96 hours. After the test paint shall not get, liver, curdle or increase in efflux time by more than 8 seconds, and there shall be no evidence of seeding. The paint shall meet the drying time requirement and shall produce dry film that is uniform in appearance and free from streaking, mottling and seediness. Further, the change in gloss value from the original shall not be more than 5 units.

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